

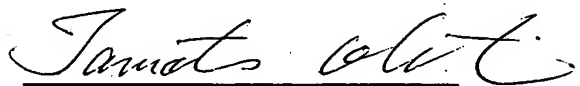
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- (1) I am fully conversant both with the Japanese and English language.
- (2) I have translated into English the Japanese Patent Application No. 2000-234048 filed on August 2, 2000. A copy of said English translation is attached hereto.
- (3) The translation is to the best of my knowledge and belief, an accurate translation of the application as filed into the English language.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief believed to be true.

Date: September 9, 2003



Tamotsu Ohtani

PATENT OFFICE
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This is to certify that the annexed is a true copy of the following application as filed with this office.

Date of Application: August 2, 2000

Application Number: Patent Application No. 2000-234048

Applicant: Idemitsu Petrochemical Co., Ltd.

May 31, 2001

Commissioner,
Patent Office

Kozo Oikawa

Certification Number 2001-3048788

Toku 2000-234048

[Document] Patent Application

[Reference No.] N00-0095

[Addressee] Commissioner of the Patent Office

[International Patent Classification] C08L 69/00

G11B 7/24

[Title of the invention]

Polycarbonate resin for optical disk substrates and the optical disk substrate

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[Payment Register No.] 041472

Toku 2000-234048

[Amount of Payment] ¥21,000

[List of Item Filed]

[Item] Specification 1

[Item] Drawing 1

[Item] Abstract 1

[General Power of Attorney No.] 0000758

[Requirement of Proof] Yes

[Document] Specification

[Title of Invention] POLYCARBONATE RESIN FOR OPTICAL DISK
SUBSTRATE AND OPTICAL DISK SUBSTRATE

[Claims]

[Claim 1] A polycarbonate resin for an optical disk substrate characterized by having a viscosity average molecular weight of from 10,000 to 17,000, an iron content of 0.2 ppm or less and a fraction of hydroxyl end groups of less than 7% by mole, and containing from 100 to 500 ppm of a releasing agent.

[Claim 2] A polycarbonate resin for an optical disk substrate as described in claim 1, which contains from 150 to 350 ppm of a releasing agent.

[Claim 3] A polycarbonate resin for an optical disk substrate described in claim 1 or 2, wherein the releasing agent is a polyhydric alcohol fatty acid ester.

[Claim 4] A polycarbonate resin for an optical disk substrate described in claim 3, wherein the polyhydric alcohol fatty acid ester is a fatty acid monoester of glycerin.

[Claim 5] An optical disk substrate comprising a polycarbonate resin for an optical disk substrate described in any of claims 1 to 4.

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] The present invention relates to a polycarbonate resin for an optical disk substrate and an optical disk substrate, and more particularly,

it relates to a polycarbonate resin that is suitable for a material of an optical disk substrate having less defects due to flash, and an optical disk substrate formed with the resin.

[0002]

[Description of the Prior Art] A polycarbonate resin is widely used as a substrate of an optical disk, such as CD, CD-ROM, MO, CD-R, CD-RW, DVD-ROM, DVD-R and DVD-RAM, owing to the transparency, the heat resistance and the low water absorbing property thereof. In general, these optical disks are often produced by such a process that a surface formed by engraving signals, such as pits and grooves, on a thin plate of nickel called as a stamper arranged in a metallic mold is transferred to a substrate formed with a polycarbonate resin through injection molding.

[0003] In the production of a substrate of an optical disk in recent years, several tens to a number exceeding one hundred of molding machines are introduced in one factory to further enhance the mass production volume efficiency, and simultaneously quality control is severely ensured. However, although there is no change of a stamper or no change in molding conditions, and the factors determining the flowability of a polycarbonate resin, such as the molecular weight, the molecular weight distribution, the glass transition temperature and the like, are stable within prescribed ranges, there are some cases where releasing failure extemporaneously occurs upon sprue cutting of a center hole of a disk substrate, and the outer circumference

of the disk substrate or fine flash formed on the outer circumference is in friction with a metallic mold due to the releasing failure to cause contamination of resin dusts, whereby the yield of the disk substrates is decreased to several percents to several tens percents. The phenomenon is generally referred to as a defect due to flash, which can be prevented by increasing an amount of a releasing agent. However, the amount of the releasing agent cannot be increased to an unnecessary level because there is a tendency of occurrence of defects on optical characteristics, particularly such a tendency that occurrence of defects of polarization and white turbidity in the polycarbonate resin is accelerated upon an accelerated aging test under constant temperature and humidity, whereby the reliability of storage stability of recorded data as an optical disk is also lowered.

[0004]

[Problem(s) to be Solved by the Invention]

The invention has been developed under the circumstances, and is to provide a polycarbonate resin that is suitable for a material of an optical disk substrate having less defects due to flash without increase of an addition amount of a releasing agent to an unnecessary level, and an optical disk substrate formed with the resin.

[0005]

[Means for Solving the Problem] As a result of various investigations made by the inventors, it has been found that

occurrence of defects due to flash is correlated with an iron amount and a fraction of hydroxyl end groups in the polycarbonate resin, and thus the first invention to the fourth invention have been completed.

[0006] That is, the summary of this invention is as follows.

1. A polycarbonate resin for an optical disk substrate characterized by having a viscosity average molecular weight of from 10,000 to 17,000, an iron content of 0.2 ppm or less and a fraction of hydroxyl end groups of less than 7% by mole, and containing from 100 to 500 ppm of a releasing agent.
2. A polycarbonate resin for an optical disk substrate as described in above 1, which contains from 150 to 350 ppm of a releasing agent.
3. A polycarbonate resin for an optical disk substrate described in above 1 or 2, wherein the releasing agent is a polyhydric alcohol fatty acid ester.
4. A polycarbonate resin for an optical disk substrate described in above 3, wherein the polyhydric alcohol fatty acid ester is a fatty acid monoester of glycerin.
5. An optical disk substrate comprising a polycarbonate resin for an optical disk substrate described in any of above 1 to 4.

[0007]

[Embodiments of the Invention]

A polycarbonate resin used in an optical disk substrate of the first invention (sometimes simply referred to as an "invention" in this chapter) will be described.

The chemical structure and the production process of the polycarbonate resin are not particularly limited, and various kinds thereof can be used. For example, an aromatic polycarbonate resin produced through a reaction between a dihydric phenol and a carbonate precursor is preferably used. The production process that can be used may be either the solution process or the molten process.

[0008] The dihydric phenol includes various kinds thereof, and preferred examples thereof include 2,2-bis(4-hydroxyphenyl)propane, bis(4-hydroxyphenyl)methane, 1,1-bis(4-hydroxyphenyl)ethane, 2,2-bis(4-hydroxy-3,5-dimethylphenyl)propane, 4,4'-dihydroxydiphenyl, bis(4-hydroxyphenyl)cyclohexane, bis(4-hydroxyphenyl)ether, bis(4-hydroxyphenyl)sulfide, bis(4-hydroxyphenyl)sulfone, bis(4-hydroxyphenyl)sulfoxide, bis(4-hydroxyphenyl)ketone, hydroquinone, resorcin, catechol and the like. Among these dihydric phenols, a bis(hydroxyphenyl)alkane, particularly 2,2-bis(4-hydroxyphenyl)propane (bisphenol A), is preferred. These dihydric phenols may be used solely or in combination of two or more of them through mixing.

[0009] As the carbonate precursor, a carbonyl halide, a carbonyl ester and a haloformate can be used. More specific examples thereof include phosgene, dihaloformate of a dihydric phenol, diphenyl carbonate, dimethyl carbonate, diethyl carbonate and the like.

[0010] With respect to the chemical structure of the

polycarbonate resin, those having a molecular chain having a linear structure, a cyclic structure or a branched structure can be used. Among these, as the polycarbonate resin having a branched structure, those produced by using, as a branching agent, 1,1,1-tris(4-hydroxyphenyl)ethane, $\alpha, \alpha', \alpha''$ -tris(4-hydroxyphenyl)-1,3,5-triisopropylbenzene, fluoroglycine, trimellitic acid, isatin bis(o-crezol) and the like are preferably used. A polyester polycarbonate resin produced by using a bifunctional carboxylic acid, such as terephthalic acid and the like, or an ester forming derivative thereof can be used as the polycarbonate resin. Furthermore, a mixture of these polycarbonate resins having various chemical structures can be used.

[0011] The viscosity average molecular weight of the polycarbonate resin is generally from 10,000 to 30,000. The viscosity average molecular weight (Mv) is a value obtained in such a manner that a viscosity of an methylene chloride solution at 20°C is measured by using an Ubbelohde viscometer to obtain a limiting viscosity $[\eta]$, and the value is calculated by the equation $[\eta] = 1.23 \times 10^{-5} Mv^{0.83}$. For the adjustment of the molecular weight of the polycarbonate resin, phenol, p-tert-butylphenol, p-tert-octylphenol, p-cumylphenol and the like are used.

[0012] A releasing agent and, depending on necessity, from 20 to 100 ppm of a phosphorous antioxidant are added to the polycarbonate resin flakes thus produced in the foregoing process,

and it is then pelletized by an extruder. It is necessary that the releasing agent in the pellets is from 100 to 500 ppm (preferably from 150 to 350 ppm).

When the releasing agent is less than 100 ppm, occurrence of defects due to flash caused by releasing failure is increased, whereas when it exceeds 500 ppm, defects of polarization and white turbidity as an optical disk are liable to be occur, and thus both cases are not preferred.

[0013] It is necessary that the pellets have a viscosity average molecular weight of from 10,000 to 17,000, an iron content is 0.2 ppm or less, and a fraction of hydroxyl end groups of less than 7% by mole. The fraction of hydroxyl end groups herein is a ratio (% by mole) of hydroxyl end groups with respect to the total end groups of the polycarbonate and is a value measured in the measuring method described later.

[0014] Among the foregoing characteristics, in order to satisfy the iron content and the fraction of hydroxyl end groups, for example, the method of purifying during the production of the bisphenol A described in JP-A-6-48970 is more effective, while not limited, in that after washing the adduct of bisphenol A and phenol with purified phenol, it is decomposed into phenol and bisphenol A, and the phenol is recycled to the reaction system.

[0015] As the releasing agent, a polyhydric alcohol fatty acid ester is preferably used, and examples thereof include a partial ester of a trihydric alcohol, such as glycerin, trimethylpropane, hexanetriol and the like, and a tetrahydric or more valence of

alcohol, such as pentaerythritol, mesoerythritol, xylitol, sorbitol and the like, with a fatty acid having a carbon number of from 10 to 30. Examples of the fatty acid include capric acid, undecanoic acid, lauric acid, tridecanoic acid, myristic acid, pentadecanoic acid, palmitic acid, margaric acid, stearic acid, nonadecanoic acid, eicosanoic acid, behenic acid and the like. Specifically, a glycerin monoester, such as glycerin monostearate, glycerin monopalmitate, glycerin monomyristate, glycerin mono laurate and the like, pentaerythritol distearate, pentaerythritol tristearate, pentaerythritol monopalmitate, pentaerythritol dipalmitate, mesoerythritol trilaurate, xylitol trilaurate, xylitol distearate, xylitol tristearate, xylitol tetrastearate and the like are used. These esters may be used singly or in combination of two or more of them.

[0016] Examples of the phosphorous antioxidant include a trialkyl phosphite, such as trimethyl phosphite, triethyl phosphite, tributyl phosphite, trioctyl phosphite, trinonyl phosphite, tridecyl phosphite, trioctadecyl phosphite, distearyl pentaerythryl diposphite, tris(2-chloroethyl)phosphite, tris(2,3-dichloropropyl)phosphite and the like; a tricycloalkyl phosphite, such as tricyclohexyl phosphite and the like; a triaryl phosphite, such as triphenyl phosphite, tricresyl phosphite, tris(ethylphenyl) phosphite, tris(butylphenyl) phosphite, tris(nonylphenyl) phosphite, tris(hydroxyphenyl) phosphite and the like; a monoalkyldiaryl phosphite, such as

2-ethylhexyldiphenyl phosphite; a trialkyl phosphate, such as trimethyl phosphate, triethyl phosphate, tributyl phosphate, trioctyl phosphate, tridecyl phosphate, trioctadecyl phosphate, distearylpennterythyl diphosphate, tris(2-chloroethyl) phosphate, tris(2,3-dichloropropyl) phosphate and the like; a tricycloalkyl phosphate, such as tricyclohexyl phosphate and the like; a triaryl phosphate, such as triphenyl phosphate, tricresyl phosphate, tris(nonylphenyl) phosphate, 2-ethylphenyl diphenyl phosphate and the like; and the like. These may be used solely or in combination of two or more kinds of them.

[0017] As described in the foregoing, it is necessary that the polycarbonate resin has, in the form of pellets, a viscosity average molecular weight of from 10,000 to 17,000, an iron content is 0.2 ppm or less, and a fraction of hydroxyl end groups of less than 7% by mole. When the iron content is too large or the fraction of hydroxyl end groups is too high, defects due to flash frequently occur.

[0018] When the viscosity average molecular weight is less than 10,000, the mechanical strength of the molded article is lowered, whereas when it exceeds 17,000, distortion remains in the molded article due to shortage of flowability upon molding, so as to lower the optical characteristics, and therefore both the cases are not preferred. The measurement methods of the iron content and the fraction of hydroxyl end groups are as follows.

(1) Iron Content

After ashing a sample, it was quantitatively analyzed by

the graphite furnace atomic absorption spectrometry.

(2) Fraction of Hydroxyl End Groups (in the case where p-t-butylphenol is used as a terminating agent)

40 mg of a sample was collected in an NMR sample tube having a diameter of 5 mm and uniformly dissolved in 0.6 mL of heavy chloroform at room temperature. It was measured for ^1H -NMR with a 500 MHz NMR produced by JEOL Ltd. (LAMBDA-500) under the following conditions.

[0019] Measurement Conditions

Pulse width: 45 degree

Pulse repetition time: 9 sec

Accumulation number: 256 times

Chemical shift standard: tetramethylsilane

Measurement temperature: room temperature

BF value: 0.15

In the NMR spectrum, the characteristic signal intensity of o-phenyl in the vicinity of the terminal OH at $\delta = 6.6$ ppm is designated as X, the characteristic signal intensity of t-butyl at $\delta = 1.3$ ppm is designated as Y, and it is obtained by the following equation.

Fraction of hydroxyl end groups (% by mole) =

$$100 \times (X/2) / ((X/2) + (Y/9))$$

The optical disk substrate of the invention is molded in the molding methods described for the first invention. Among these, the injection molding method using a stamper is preferred.

[0020]

[Example] Next, although an example and the example of comparison explain this invention still more concretely, this invention is not limited at all by these examples.

(Example 1)

(1) Production of Bisphenol A

According to the production scheme of bisphenol A shown in Fig. 7, bisphenol was produced by a continuous process. Phenol, acetone and ethylmercaptan were continuously introduced to a reactor 1 having 600 g of a cationic resin filled therein at rates of 4,600 g/hr, 280 g/hr and 16 g/hr, respectively, while maintaining the temperature at 75°C. A reaction mixture 2 was introduced to a concentrating step 3 and concentrated under the conditions of 165°C and 53.3 kPa to make a concentration of bisphenol A of 30% by mass, and a concentrated liquid 4 was then introduced to a crystallizing step 5 and subjected to a cooling treatment under conditions of 45°C, whereby an adduct of bisphenol A and phenol was crystallized. A slurry 6 of the adduct was then transported to a separating step 7 and separated into adduct crystals and a mother liquid. The adduct crystals 8 was washed with purified phenol in an amount 0.5 time (by weight) the amount of the wet cake, and the adduct crystals 10 thus washed was subjected to recrystallization and then transported to a adduct decomposition step 11. In the adduct decomposition step, the adduct was decomposed under conditions of 2.6 kPa and 170°C, whereby the most amount of phenol was distilled off. The whole

amount of the distilled phenol 12 was recycled to the reactor 1. On the other hand, residual phenol was removed substantially completely by steam stripping from bisphenol A 13 taken out from the decomposition step 11, so as to obtain bisphenol A as a product.

[0021]

(2) Preparation of Polycarbonate Oligomer

To 400 L of a 5% by mass sodium hydroxide aqueous solution, 60 kg of the bisphenol A produced in the foregoing manner was dissolved to prepare a sodium hydroxide solution of bisphenol A.

Then, the sodium hydroxide solution of bisphenol A maintained at room temperature was introduced at a flow rate of 138 L/hr and methylene chloride was introduced at a flow rate of 69 L/hr to a tubular reactor having an inner diameter of 10 mm and a tube length of 10 m through an orifice plate, to which phosgene was blown at a flow rate of 10 kg/hr, so as to carry out a reaction for 3 hours in a continuous manner. The tubular reactor used herein had a double tube structure, and cooling water is passed through the jacket part to maintain the discharging temperature of the reaction liquid to 25°C.

The pH of the discharged liquid was adjusted to from 10 to 11. The thus resulting reaction liquid was allowed to stand to separate and remove an aqueous phase, whereby a methylene chloride phase (220 L) was collected to obtain a polycarbonate oligomer solution.

[0022]

(3) Production of Polycarbonate

To 10 L of the polycarbonate oligomer solution obtained in the item (2), 118 g of p-t-butylphenol was dissolved, to which a sodium hydroxide aqueous solution (NaOH: 75 g, water: 1 L) and 1.17 mL of triethylamine were added, and stirred at 300 rpm at ordinary temperature for 30 minutes. 8 L of methylene chloride and a sodium hydroxide aqueous solution of bisphenol A (bisphenol A: 607 g, NaOH: 320 g, water: 5 L) were then added thereto and stirred at 500 rpm at ordinary temperature for one hour. Thereafter, 5 L of methylene chloride was added and stirred at 500 rpm at ordinary temperature for 10 minutes. After termination of stirring, standing separation was carried out to obtain an organic phase. The organic phase was alkali-washed with 5 L of a 0.03 N sodium hydroxide aqueous solution, washed with 5 L of 0.2 N hydrochloric acid, and washed with 5 L of water (twice) in this order, and then methylene chloride was distilled off to obtain polycarbonate in a flake form. The resulting polycarbonate flakes were dried in vacuum at 120°C for 48 hours, so as to obtain polycarbonate flakes having a viscosity average molecular weight of 14,500. About 50 kg of polycarbonate flake was obtained in the similar operations.

[0023]

(4) Production of Pellets

To the polycarbonate in a flake form obtained in the item (3), 300 ppm of glycerin monostearate as a releasing agent and

40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a viscosity average molecular weight of 14,200, an iron content of 0.1 ppm, a fraction of hydroxyl end groups of 5.4% by mole and a releasing agent amount of 250 ppm.

[0024]

(5) Molding and Examination

The pellets obtained in the item (4) were fed to an injection molding machine (DISK5, produced by Sumitomo Heavy Industries, Ltd.), and 600 pieces of disk substrates having a diameter of 130 mm and a thickness of 1.2 mm under the following conditions.

Cylinder temperature: 325°C

Metallic mold temperature: 90°C (stamper side)/85°C

Stamper: for CD-ROM

The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 4.5%. The disk substrates were subjected to accelerated deterioration under constant temperature and humidity of 90°C and 90% for 300 hours, and the block error rate was 6 when they were measured with an electric characteristics inspection machine.

[0025]

(Comparative Example 1)

(1) Production of Bisphenol A

According to the production scheme of bisphenol A shown in Fig. 2, bisphenol was produced by a continuous process. That

is, the same procedures as in the item (1) of Example 1 were carried out to produce bisphenol A except that the distilled phenol 12 was not recycled to the reactor 1 but used instead of the purified phenol in the adduct washing step 9.

(2) Preparation of Polycarbonate Oligomer

A polycarbonate oligomer solution was obtained in the same manner as in the item (2) of Example 1 by using the bisphenol A produced in the item (1) of Comparative Example 1.

(3) Production of Polycarbonate

Polycarbonate flakes having a viscosity average molecular weight of 14,300 were obtained in the same manner as in the item (3) of Example 1 by using the polycarbonate oligomer solution produced in the item (2) of Comparative Example 1

[0026]

(4) Production of Pellets

To the polycarbonate in a flake form obtained in the item (3), 300 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a viscosity average molecular weight of 14,100, an iron content of 0.3 ppm, a fraction of hydroxyl end groups of 7.5% by mole and a releasing agent amount of 250 ppm.

(5) Molding and Examination

600 pieces of disk substrates having a diameter of 130 mm and a thickness of 1.2 mm were produced by using the pellets obtained in the item (4) in the same manner as in the item (5)

of Example 1.

The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 16%.

[0027]

(Comparative Example 2)

(1) Production of Bisphenol A

Bisphenol A was produced in the same manner as in the item (1) of Example 1.

(2) Preparation of Polycarbonate Oligomer

A polycarbonate oligomer solution was obtained in the same manner as in the item (2) of Example 1.

(3) Production of Polycarbonate

Polycarbonate flakes were obtained in the same manner as in the item (3) of Example 1.

[0028]

(4) Production of Pellets

To the polycarbonate in a flake form obtained in the item (3), 600 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a viscosity average molecular weight of 14,200, an iron content of 0.1 ppm, a fraction of hydroxyl end groups of 5.4% by mole and a releasing agent amount of 530 ppm.

(5) Molding and Examination

600 pieces of disk substrates having a diameter of 130 mm

and a thickness of 1.2 mm were produced by using the pellets obtained in the item (4) in the same manner as in the item (5) of Example 1.

The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 4%. After the disk substrates were subjected to accelerated deterioration under constant temperature and humidity of 90°C and 90% for 300 hours, the block error rate was 28 when they were measured with an electric characteristics inspection machine.

[0029]

(Comparative Example 3)

(1) Production of Bisphenol A

Bisphenol A was produced in the same manner as in the item (1) of Example 1.

(2) Preparation of Polycarbonate Oligomer

A polycarbonate oligomer solution was obtained in the same manner as in the item (2) of Example 1.

(3) Production of Polycarbonate

Polycarbonate flakes were obtained in the same manner as in the item (3) of Example 1.

[0030]

(4) Production of Pellets

To the polycarbonate in a flake form obtained in the item (3), 30 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous

antioxidant were added, and then pelletized with an extruder. The pellets had a viscosity average molecular weight of 14,200, an iron content of 0.1 ppm, a fraction of hydroxyl end groups of 5.4% by mole and a releasing agent amount of 20 ppm.

(5) Molding and Examination

600 pieces of disk substrates having a diameter of 130 mm and a thickness of 1.2 mm were produced by using the pellets obtained in the item (4) in the same manner as in the item (5) of Example 1.

The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 17%.

[0031]

[Effect of the Invention] According to this invention, the optical disk substrate by which the defect in a flash comes to fabricate the polycarbonate resin constituent and this resin constituent which were suitable as a material of a few optical disk substrate can be offered, without increasing the addition of a release agent more than required.

[BRIEF DESCRIPTION OF THE DRAWINGS]

Fig. 1 shows a diagram of a production scheme of bisphenol A in Example 1, Comparative Example 2 and Comparative Example 3.

Fig. 2 shows a diagram of a production scheme of bisphenol A in Comparative Example 1.

[DESCRIPTION OF THE SYMBOLS IN THE DRAWINGS]

In Figs. 1 and 2, symbol 1 denotes a reactor, symbol 3 denotes a condensation step, symbol 5 denotes a crystallizing step, symbol 7 denotes a separating step, symbol 9 denotes a washing step, symbol 11 denotes an adduct decomposition step, symbol 12 denotes distilled phenol, and symbol 13 denotes bisphenol A.

[Document]

Abstract

[Abstract]

[Objects]

To obtain a polycarbonate resin which is suitable as the material for optical disk substrates with reduced defects due to flashes without unnecessarily increasing the amount of a mold release agent to be added, and an optical disk substrate composed of the resin.

[Means for Solving the Problems]

A polycarbonate resin for an optical disk substrate characterized by having a viscosity average molecular weight of from 10,000 to 17,000, an iron content of 0.2 ppm or less and a fraction of hydroxyl end groups of less than 7% by mole, and containing from 100 to 500 ppm of a releasing agent.

The optical disk substrate is composed of the resin.

[Selected Drawings] none

Fig. 1

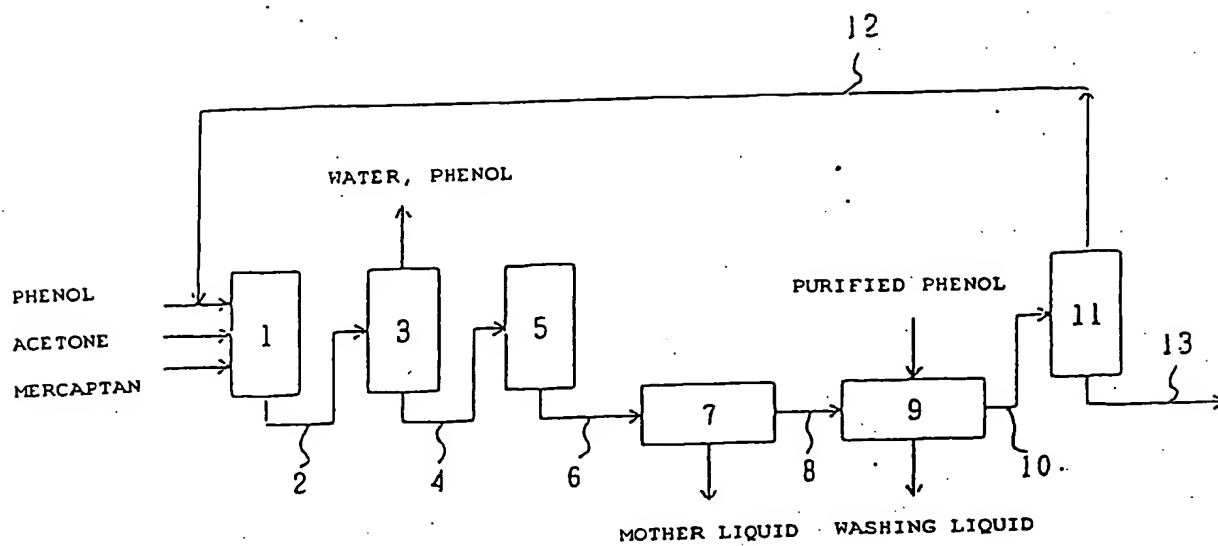


Fig. 2

